भारतीय मानक
Indian Standard

IS 10532 (Part 4) : 2019

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भाग 4 फॉस्फेट एस्टर प्रकार

(पहला पुनरीक्षण)

Fire Resistant Hydraulic Fluids — Specification

Part 4 Phosphate Esters Type

(First Revision)

ICS 75.100

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on recommendation of the Lubricants and their Related Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The use of fire-resistant hydraulic fluids is increasing due to a growing awareness of the dangers inherent in using mineral oil for applications where there is fire risk. There are mainly four types of fire resistant hydraulic fluid (FRHF) namely, dilute emulsions, invert emulsions, water glycols and synthetic fluids. In synthetic fluids, FRHF based on phosphate esters are more commonly used. These can be straight synthetic fluids in pure phosphate esters or synthetic base fluids compounded with hydrocarbon fractions. They have high film strength and good lubrication performance. Because of high thermal stability, they can operate at temperatures up to 150°C depending upon the system design. However, non-metal components of hydraulic systems, such as seals, hoses and paints commonly used in petroleum oil systems, generally speaking, are not compatible with these synthetic fluids. Special seals and others are, therefore, required to be used in systems using these fluids.

This Indian Standard is published in four parts. The other parts in this series are:

Part 1 Dilute emulsions for powered supports.

Part 2 Invert emulsions (water-in-oil) type

Part 3 Water glycol type

Selection and used of the fire-resistant hydraulic fluids are covered in IS 10531 ‘Code of practice for selection and use of fire resistant hydraulic fluids’ while determination of their fire resistant characteristic is given in IS 7895 ‘Tests for fire resistant characteristics of hydraulic fluids used in mining machinery’.

This standard was originally published in 1983. In this first revision of the standard, one more viscosity grade of VG 68 is included. The designation of various grades is redesignated as HFDR instead of HF-D. Further requirements of flash point, auto-ignition temperature, kinematic viscosity, pour point, foaming characteristics, air release properties and total oxidation products (TOP) are modified for various grades. The requirement of foaming stability is merged with foaming tendency. Two amendments issued to the earlier version were considered in this revision.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*).’ The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FIRE RESISTANT HYDRAULIC FLUIDS — SPECIFICATION

PART 4 PHOSPHATE ESTERS TYPE

(First Revision)

1 SCOPE

This standard (Part 4) prescribes the requirements and methods of sampling and tests for fire-resistant hydraulic fluids, phosphate esters type, suitable for use in hydraulic control systems.

2 REFERENCES

The following standards contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1447 [P : 1] : 2000	Petroleum and its products — Methods of sampling: Part 1 Manual sampling (*first revision*)
1448	Methods of test for petroleum and its products
[P : 2] : 2007/ ISO 6619 :1988	Petroleum products and lubricants — Neutralization number
[P : 10/Sec 2] : 2013/ ISO 3016 : 1994	Cloud point and pour point, Section 2 Determination of pourpoint (*second revision*)
[P : 25/Sec 1] : 2018/ ISO 3104 : 1994	Transparent and opaque liquids, Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
[P : 32] : 1992	Density and relative density (*second revision*)
[P : 40] : 2015/ ISO 3733 : 1999	Water by distillation (*third revision*)
[P : 67] : 1982	Foaming characteristics of lubricating oils (*first revision*)
[P: 69] : 2013/ ISO 2592 : 2000	Determination flash and fire points — Cleveland open cup method (*first revision*)
[P : 87] : 1979	Auto ignition temperature of liquid petroleum products
[P : 95] :1980	Determination of demulsification number of lubricating oils
[P : 96] :1980	Rust-preventing characteristics of steam-turbine oil in the presence of water
[P : 102] :1981	Determination of air release value
[P :170] : 2018/ ISO 20623 : 2003	Determination of the extreme-pressure and anti-wear properties of fluids — Four ball method european conditions
7895 : 1975	Tests for fire — Resistant characteristics of hydraulic fluids used in mining machinery

3 GRADES

The material shall be in four viscosity grades namely VG 22, VG 32, VG 46 and VG 68 designated as HFDR- 22, HFDR-32, HFDR-46 and HFDR-68.

4 REQUIREMENTS

4.1 Description

The material shall be a clear fluid, free from foreign matter, sediment and visible impurities. It shall not contain any ingredients injurious to persons using or handling it.

4.2 Composition

The material shall be a blend of phosphate esters with additives necessary for desirable antioxidant, anti-rust and antifoaming properties.

4.3 The material shall also comply with the requirements prescribe in Table 1, when tested according to the appropriate methods specified in col 7 of the table.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier. Galvanized drums/barrels shall not be used for packing these fluids.

5.2 Marking

5.2.1 The containers shall be securely closed and marked with the following information:

a) Name, type and grade of material;
b) Manufacturer's name, initials or trade-mark, if any;
c) Net mass of material;
d) Identification in code or otherwise to enable the lot of consignment or manufacture to be traced back from records;
e) Instructions for use; and
f) Any other statutory requirements.

5.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.2.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part : 1).

6.1 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample meet the relevant specification requirements.

7 STORAGE

The material shall be stored and handled, strictly in accordance with the supplier's instructions.

8 SAFETY PRECAUTIONS

8.1 While handling these fluids the following safety precautions shall be observed:

a) The fluid does not cause skin irritation ordinarily but wearing of protective clothing is recommended.
b) Continued exposure to the fumes is to be avoided. Breathing vapors from heated or burning product to be avoided.
c) Smoking, eating or drinking shall be prohibited when these fluids are being handled.

Table 1 Requirements for Fire-Resistant Hydraulic Fluids-Phosphate Esters Type

(*Clause* 4.3)

Sl No.	Characteristics	Requirements				Methods of Test
		Grade HFDR 22	Grade HFDR 32	Grade HFDR 46	Grade HFDR 68	Refer to Annex / [P :] of IS 1448 / ASTM
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Flash point, (cleve land open cup) °C, *Min*	180	200	215	220	[P : 69]
ii)	Fire point, (cleve land open cup) °C, *Min*	290	290	290	290	[P : 69]
iii)	Auto-ignition temperature, °C, *Min*	580	530	500	500	[P : 87]
iv)	Relative density at 15/15, *Max*	1.25	1.25	1.25	1.25	[P : 32]
v)	Kinematic viscosity, cSt					[P : 25/ Sec 1]
	a) At 40°C	19.8 - 24.2	28.8 - 35.2	41.4 - 50.6	61.2 - 74.8	
	b) At 0°C, *Max*	450	900	1700	Report	
	c) At 100°C, *Min*	2.5	3.0	4.0	6.0	
	d) Minimum pumping temp °C , *Max*	-5	0	5	10	
vi)	Pour point, °C, *Max*	-21	-12	-6	-3	[P : 10]
vii)	Foaming characteristics:					
	Foaming tendency/ stability					[P : 67]
	1) Foam volume, ml/ml at 24°C, *Max*	300/10	300/10	300/10	300/10	
	2) Foam volume, ml/ml at 93°C, *Max*	300/10	300/10	300/10	300/10	
	3) Foam volume, ml/ml at 24°C after test at 93°C, *Max*	300/10	300/10	300/10	300/10	
viii)	Demulsification number, seconds, *Max*	300	300	600	600	[P : 95]
ix)	Air release properties, minutes to 0.2 percent volume air content at 50°C, *Max*	10	12	15	25	[P : 102]
x)	Total acidity, mg KOH/g, *Max*	0.2	0.2	0.2	0.2	[P : 2]
xi)	Stability tests:					
	a) *Oxidation and Thermal*					Annex A
	1) Total oxidation products (TOP), percent, *Max*	0.3	0.3	0.3	0.3	
	2) Sludge , percent, of TOP, *Max*	50	50	50	50	
	b) *Hydrolytic and Corrosion*					
	1) Total oxidation products (TOP), percent *Max*	0.8	0.8	0.8	0.8	
	2) Sludge , percent of TOP, *Max*	50	50	50	50	
	3) Corrosion, mg, *Max*	5.0	5.0	5.0	5.0	
xii)	Rust Preventive characteristics	Shall pass the test after 20 h				[P : 96] (Method A)
xiii)	Water content, percent by weight, *Max*	0.2	0.2	0.2	0.2	[P : 40]
xiv)	Four ball test , 40 kg, 1200 rev/min, 75 ± 2°C for 1 h, mm, *Max*	To report	To report	To report	To report	[P : 170]/D 4172
xv)	Fire resistant characteristics:	Shall pass the tests A, B and C				7895
	a) Auto-ignition temperature					
	b) Temperature pressure spray ignition test					
	c) Flame propagation test					

ANNEX A

[*Table* 1, *Sl No.* (xi)]

TEST FOR OXIDATION AND HYDROLYTIC STABILITY AND CORROSION POTENTIAL

A-1 SCOPE

This method is comparatively short test to measure the resistance to oxidation and hydrolysis under specified conditions of new phosphate ester fire-resistant fluids. It also measures the corrosivity of the fluid and its degradation products in the presence of water.

A-2 OUTLINE OF THE METHOD

Oxygen is passed for 164 h through a sample A of the fluid, and another sample B of the fluid with added solid metal catalysts (iron and copper) and water whilst maintained at 120°C. The volatile acid products, the acidity of the fluid; the sludge and the sludge insoluble in organic solvents are determined, where sample A is used for oxidation and thermal stability, and sample B for hydrolytic stability and corrosivity.

A-3 APPARATUS

A-3.1 Oxidation Tubes

Manufactured from borosilicate or neutral glass having the dimensions shown in Fig. 1.

A-3.2 Heating Bath

An aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the desired number of oxidation tubes at the required temperature of 120 ± 0.5°C (*see* Fig. 2). This temperature shall be read on a thermometer having a range of 98 – 152°C and least count of 0.2°C (*see* Note) inserted in a test tube to within 5 mm from the bottom; this test tube shall be filled with oil up to the immersion line of the thermometer and placed in the heating bath. The temperature of the upper surface shall be kept at 60 ± 0.5°C. Measure this temperature by the use of a thermometer in a drilled aluminium block (*see* Fig. 2); the surfaces of this block, other than that against the upper surface of the heating bath, are protected by suitable insulation. This block should be placed as near to the holes as practicable and within the area of the aluminium heater block. When using an aluminium block heater the test tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube. In the case of oil baths the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath. For both types of heating bath the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In case of a slackness a 25 mm diameter O-ring may be placed round the tube and pressed against the heater surface.

NOTE — Thermometer of type IP 81C may also be used.

A-3.3 Filtering Crucibles

Gooch type crucibles with sintered filter disc of Grade 4 porosity (5 to 15 microns), 35 ml capacity.

A-3.4 Porcelain Crucibles

50 ml capacity.

A-3.5 Soap Bubble Flowmeter

For checking the oxygen flow rate (*see* Fig. 3).

A-3.6 Burette

Volume 10 ml with graduations of 0.01 ml.

A-3.7 Thermometers

Conforming to temperature ranges of:

a) 98 to 152°C, having least count 0.2°C, and
b) -5 to +110°C, having least count 0.5°C or others of suitable range and equal or greater accuracy.

NOTE — Thermometers of specifications IP 81C and IP 15C may also be used.

A-3.8 Absorption Tubes

These are similar to the oxidation tubes (*see* **A-3.1**) but with the bubbling tube modified as in Fig. 4.

A-4 REAGENTS AND MATERIALS

A-4.1 Catalysts

a) *Copper wire* — 1.04 ± 0.01 mm diameter, annealed, plain.
b) *Low Metalloid Steel Wire (Thermocouple Quality)* — 1 mm diameter.

A-4.2 Oxygen — commercial product obtained from liquid air (minimum purity 99.4 percent). The oxygen shall be dried by passing through a suitable desiccant. A 10 litre flask, acting as a surge vessel, smoothes the oxygen flow, excess of which bubbles through mineral oil contained in a test tube. The method used to regulate the oxygen flow is left to the discretion of the operator, but the flow rate shall be checked by use of the soap bubble flow meter (*see* Fig. 3) connected after the

absorption vessel.

A-4.3 Alkali Blue Solution — 2 g/100ml.

A-4.4 Phenolphthalein Solution — 1 g/100 ml alcoholic solution.

A-4.5 *n*-Heptane — same as that used as a reference fuel in determining the octane number of gasoline.

A-4.6 Hydrochloric Acid — 0.1 N aqueous solution.

A-4.7 Potassium Hydroxide — 0.1 N alcoholic solution.

A-4.8 Toluene — Pure, Sulphur-free.

A-4.9 Chloroform

A-4.10 Isopropyl Alcohol

A-4.11 Methyl Alcohol

A-5 PREPARATION OF APPARATUS

A-5.1 Cleaning the Test Tubes

The oxidation and absorption tubes shall be chemically cleaned. A satisfactory method of cleaning is to wash with acetone, followed by distilled water. Drain and then soak in concentrated sulphuric acid for a minimum of 16 h. Drain and complete removal of the acid by washing, first with tap water, then with distilled water. Dry the tubes in an air oven at 105 to 110°C for at least 3 h, and then allow them to cool to room temperature in a desiccators in which they are kept until they are used.

A-6 PROCEDURE

A-6.1 Weigh sample A 25 g and sample B 10 ± 0.5 g of the fluid into two oxidation tubes respectively. Clean 300 mm each of the copper and iron wire with absorbent cloth wet with n-heptane, followed by abrasion with No. 100(00) silicon carbide cloth until a fresh metal surface is exposed. Wipe with dry absorbent cotton until all loose particles of abrasive and metal have been removed. In subsequent operations handle the wires with absorbent cotton (gloves) or tweezers to prevent them coming into contact with the skin. Wind the wires simultaneously alongside each other on a threaded mandrel. Lift the ends of the wires from the slot in the end of the mandrel and twist together for approximately three turns. Adjust the length of the coil to 20 mm. Store in petroleum spirit until insertion in tube B. Put tubes A and B into the heating bath at 120 ± 0.5°C.

A-6.2 Connect the oxidation tubes to the absorption tubes into which has been placed 25 ml of neutral distilled or deionized water and 5 to 6 drops of phenolphthalein solution (*see* Note).

NOTE — To avoid evaporation of water the absorption tube shall be protected from the heating bath by insulation.

A-6.3 Connect and adjust the oxygen flow at 1.0 ± 0.1 l/h which shall be checked daily. After 1 h slightly raise the drechsel head of tube B and add 0.5 ml distilled water, from a fine bore pipette, through the gap between the socket and cone. Repeat the addition of water after 24, 48, 72 and 96 h.

A-6.4 After 164 h, stop the oxygen flow, disconnect the oxidation and absorption tubes and remove the oxidation tubes from the bath.

A-6.5 Treat the absorption tubes as follows:

A-6.5.1 *Volatile Acids*

As quickly as possible after the test, titrate the water in the absorption tube with the alcoholic potassium hydroxide solution.

A-6.6 Treat the oxidation tube as follows:

A-6.6.1 *Sludge Determination*

Cool the sample of 25 g of artificially aged oil in the dark for 1 h and then pour it into a conical flask of 500 ml capacity, fitted with a ground glass stopper. Use 300 ml, a mixture of equal volumes of n-heptane (*see* Note) and toluene to recover the oil adhering to the test tube and oxygen lead-in tube and add the washings to the oil in the flask.

NOTE — If the normal heptanes recovered from previous test is used it shall be acid-free and comply with the original specification (*see* **A-4.5**).

A-6.6.1.1 Allow the mixture to stand in the dark for 24 h at a temperature of 20 ± 2°C, then filter through the filtering crucible previously dried to constant mass.

A-6.6.1.2 At the start of filtering only a small pressure drop should be used to prevent the sludge passing through the filter. Cloudy filtrates should be passed through a second time.

A-6.6.1.3 Carefully remove all traces of oil by repeated washing of the sludge with a mixture of equal volumes of normal heptanes and toluene. The total volume of the solvent used for the washing of the sludge shall be 150 ml. Dry the crucible containing the sludge at 110°C to constant mass. The filtrate shall be used for the determination of soluble acidity.

A-6.6.1.4 Dissolve any sludge adhering to the test tube and to the oxygen lead in tube in small quantities of chloroform (a total of 30 ml) and transfer the solution into a tarred porcelain crucible. After the evaporation of the chloroform dry at 110°C to constant mass.

A-6.6.2 *Soluble Acidity*

Collect the heptane / toluene solution obtained after filtering off the sludge in a 500 ml measuring flask and make up to the mark with a mixture of equal volumes of n-heptane and toluene. Make three determinations

of the neutralization value on 100 ml samples of the n-heptane / toluene / oil solution.

A-6.6.2.1 Immediately before use prepare the titration solvent as follows:

Add 2 ml of the alkali blue solution to 100 ml of a mixture of 60 ml of toluene and 40 ml of isopropyl alcohol containing 5 percent of water. Neutralize the mixture of 60 ml alcoholic potassium hydroxide solution to give a red color comparable to that of a 10 percent solution of cobalt nitrate Co $(NO_3)_2$. $6H_2O$, and this color shall persist for at least 15 s. Add this neutralized solvent with swirling, to 100 ml of the heptanes/toluene solution then titrate with the alcoholic potassium hydroxide at a temperature not exceeding 30°C.

A-6.7 Insoluble and Inorganic Material from Tube (B)

A-6.7.1 After drying and weighing the sludge in the filter and porcelain crucibles from tube (B) proceed as follows:

Extract the sludge in the porcelain crucible with successive quantities of warm chloroform and methyl alcohol, approximately 50 ml of each. Transfer to the filter crucible and vacuum filter. Dry the filter crucible for 1 h at 105 to 110°C. Cool for 1 h and weigh. Note that to repeat results for low sludge and insoluble contents (for example < 0.02 percent), clean solvents shall be used. While solvents are normally sufficiently clean it may be advisable to pre-filter all solvents through a No. 4 filter disc.

A-7 CALCULATION AND REPORT

A-7.1 Volatile Acidity

$$(\text{VA, mg KOH/g}) = \frac{A \times 56.1 \times N}{25} \quad \text{for tube (A)}$$

$$\text{and} = \frac{A \times 56.1 \times N}{10} \quad \text{for tube (B)}$$

A-7.2 Soluble Acidity

$$(\text{SA, mg KOH/g}) = \frac{A \times 56.1 \times N}{5} \quad \text{for tube (A)}$$

$$\text{and} = \frac{A \times 56.1 \times N}{2} \quad \text{for tube (B)}$$

where,

A = the volume of 0.1 N alcoholic potassium hydroxide solution necessary to neutralize the n-heptane / toluene solution, and

N = the normality of the alcoholic KOH solution used.

A-7.3 Total acidity (TA) = VA + SA

A-7.4 Total sludge (D percent) = (a + b) × 4 for tube (A)

and = (a + b) × 10 for tube (B)

where,

a = the mass of sludge insoluble in toluene/ n-heptane, in grams; and

b = the mass of sludge recovered by chloroform, in grams.

A-7.5 Total Oxidation Products

$$(\text{TOP percent}) = D + \frac{TA \times 100 \times 0.18}{1000 \times 0.0561}$$

A-7.6 Insoluble and inorganic material for tube B = mass of material insoluble in chloroform and methyl alcohol in grams.

A-8 PRECISION

An indication of the precision of this method can be obtained from the following (within laboratory reproducibility).

A-8.1 Oxidation and Thermal Stability

A-8.1.1 *Total Oxidation Products*

Duplicate results obtained by the same operator in the same laboratory on tests carried out at different times should be considered suspect if they differ by more than 28 percent of their mean for results in the range 0.05 to 3.0 percent.

A-8.2 Hydrolytic Stability

A-8.2.1 *Total Oxidation Products*

Duplicate results obtained by the same operator in the same laboratory on tests carried out at different times should be considered suspect if they differ by more than 28 percent of their mean for results in the range 0.13 to 3 percent.

A-8.2.2 *Corrosion*

Duplicate results obtained by the same operator in the same laboratory on tests carried out at different time should be considered suspect if they differ by more than 112 percent of their mean for results in the range 0.0 to 5.0 mg insoluble and inorganic deposits.

ANNEX B

BIBILOGRAPHY

ASTM	*Title*
D 4172- 18	Standard Test Method for Wear Preventive Characteristics of Lubricating Fluid (Four-Ball Method)

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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